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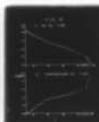
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CHEMICAL GENERATION OF O2((1) DELTA SUB 6) I.(U)
MAY 79 L E MYERS, L P DAVIS, C J DYMEK

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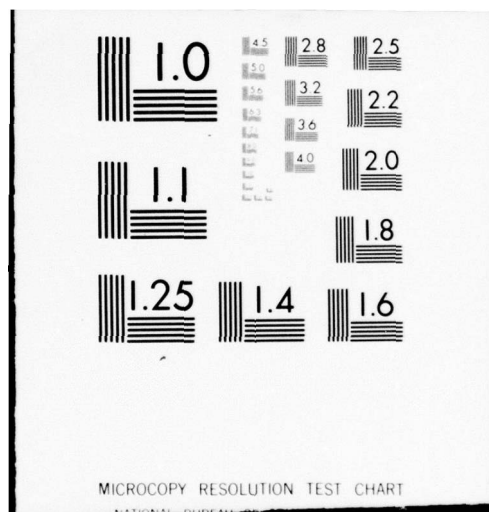
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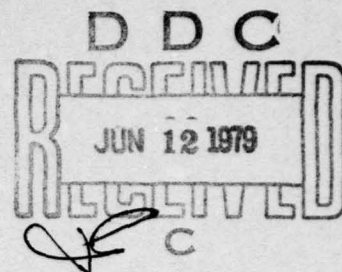


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FRANK J. SEILER RESEARCH LABORATORY

FJSRL TECHNICAL REPORT-79-0005

MAY 1979

CHEMICAL GENERATION OF $O_2(^1\Delta_g)$ I.

LT L. E. MYERS
CAPT L. P. DAVIS
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PROJECT 2303

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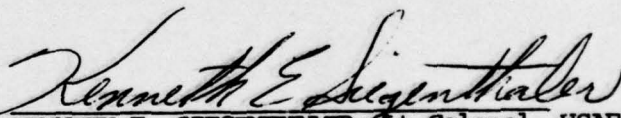
This document was prepared by the Molecular Dynamics Division, Directorate of Chemical Sciences, Frank J. Seiler Research Laboratory, United States Air Force Academy, Colorado. The research was conducted under Project Work Unit Number 2303-F4-01, "Chemiluminescent Gas Phase Reactions". Lt Lee E. Myers was the Project Scientist in charge of the work.

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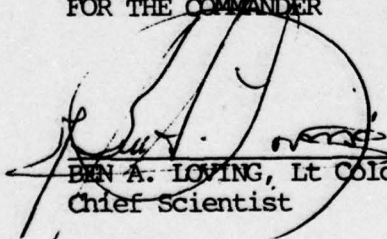
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CHEMICAL GENERATION OF $O_2(^1\Delta_g)$ I.

By

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MAY 1979

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PREFACE

This interim report documents work done under Work Unit 2303-F4-01, Chemiluminescent Gas Phase Reactions, between 1 December 1977 and 18 December 1978. Work is continuing and will be documented in future interim reports and/or a final report as events warrant. The authors wish to thank B. J. Darcy for typing the manuscript and F. C. Kibler, Jr. for his assistance and skill in preparing and maintaining the glass generator vessel.

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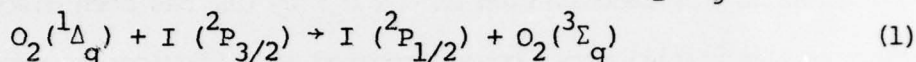
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I. INTRODUCTION

The $O_2(^1\Delta_g)/I$ chemical laser system has evolved rapidly during the last year in terms of both the chemical generator used to produce the $O_2(^1\Delta_g)$ and the actual achievement of lasing¹. The basis of this electronic transition laser system is energy transfer from $O_2(^1\Delta_g)$ to I:



The key to successful lasing of this system is the generation of oxygen flows with $O_2(^1\Delta_g)/O_2(\text{total})$ ratios well in excess of 0.17. This is most successfully done by reacting Cl_2 with a basic hydrogen peroxide solution and pumping the oxygen product away from the reactor as quickly as possible. This type of generator was first proposed and developed for iodine laser pumping at FJSRL under a previous work unit^{2,3} and then modified and improved at AFWL, where successful lasing on the 1.315μ transition of I has been observed¹.

Previous work at FJSRL has focused on deactivation of $O_2(^1\Delta_g)$ and determination of HCl content in the chemical generator exhaust⁴. The development of a new chemical generator modeled on the AFWL design has resulted in the emphasis shifting to the determination of the reaction mechanism and optimization of % $O_2(^1\Delta_g)$ in the generator output. The new generator has proved to be a reliable, consistent producer of $O_2(^1\Delta_g)$ in percentages of up to 35%. Thus it can be used as a test instrument to determine effects of changes in operating conditions on the $O_2(^1\Delta_g)$ production and to study the reaction mechanism.

The effects of various reaction parameters have been studied both qualitatively and quantitatively. These parameters have included pressure,

flow rate, reactor configuration, and trap temperature. The reactor parameters routinely monitored include pressure, flow rate, temperature, and pH. Trends in the $O_2(^1\Delta_g)$ production are presented and discussed in this report.

The study of deactivation of $O_2(^1\Delta_g)$ by Cl_2 has been discontinued because the initial upper bound reported in a previous report⁴ indicated that the deactivation would not be a problem in the reactor system. The determination of HCl in the exhaust was discontinued because of experimental difficulties. A new on-line mass spectrometer is on order and will be used to determine species in the reactor output.

II. EXPERIMENTAL

Qualitative Observations

A series of qualitative observations were made of the parametric effects of the liquid reactant concentrations, temperature, pressure, and Cl_2 flow rate upon $O_2(^1\Delta_g)$ production. The intensity of the $[O_2(^1\Delta_g)]_2$ dimol emission ($\lambda = 0.634 \mu m$) was visually observed as an indication of the relative concentration of $O_2(^1\Delta_g)$ molecules. Similarly, visual observations were made of the $I_2(B \rightarrow X)$ emission, resulting from the addition of I_2 to the product stream. Both the dimol and iodine emission intensities were assumed proportional to the $O_2(^1\Delta_g)$ yield. A schematic diagram of the experimental set-up is given in Fig. 1.

Quantitative Studies - General Description

A schematic diagram of the overall experimental apparatus is given in Fig. 2 and a more detailed illustration of the chemical generator is given in Fig. 3. In the experiments reported here, the reaction mixture was

generally a 1:1 by volume mixture of 90% H_2O_2 (FMC Corp.) and 6.0 M NaOH. The Cl_2 ⁵ was used without further purification, and its mass flow rate into the reactor was monitored using a Matheson thermal conductivity type flow meter. $\text{O}_2(^1\Delta_g)$ and $\text{O}_2(^3\Sigma_g)$ outputs from the reactor were monitored by a Varian Model 4502 ESR spectrometer. The $K = 3$, $J = 4$, $M = 3 \rightarrow 4$ transition of $\text{O}_2(^3\Sigma_g)$ ⁶ (the so-called "A-line") and one of the center quadruplets of the $\text{O}_2(^1\Delta_g)$ spectrum were used to monitor the $\text{O}_2(^3\Sigma_g)$ and $\text{O}_2(^1\Delta_g)$ quantities. The total pressure from the generator was measured at the ESR cavity by a Baratron capacitance-type manometer with a 10-torr head. The $\text{O}_2(^3\Sigma_g)$ ESR measurements were calibrated by flowing O_2 gas through the cavity under the same flow conditions as obtained for the reactor output. A calibration plot for the double integral of the $\text{O}_2(^3\Sigma_g)$ ESR spectrum versus the pressure measured at the cavity is shown in Fig. 4. The $\text{O}_2(^1\Delta_g)$ pressures were determined by use of the calibration plot and a factor relating the theoretical intensities of $\text{O}_2(^3\Sigma_g)$ and $\text{O}_2(^1\Delta_g)$ spectra⁴. The double integration was performed using a Hewlett-Packard Model 9864A digitizer to read the spectra. The digitized spectra were input into a Hewlett-Packard Model 9830A desk calculator programmed to compute the double integrals and convert them to $\text{O}_2(^1\Delta_g)$ and $\text{O}_2(^3\Sigma_g)$ pressures.

The temperature in the reaction mixture was monitored using a Chromel-Alumel thermocouple input into a 400A Doric Trendicator. The pH of the reaction mixture was monitored using a Markson pHexiMark electrode input into a Beckman SS-2 pH meter which included an expanded scale capability. The electrode gives valid pH readings over the temperature range -5°C to

100°C according to the manufacturer. Since we were primarily interested in trends in pH in this work, the corrections to pH readings made with glass electrodes in H_2O_2 solutions suggested by Kolczynski *et al*⁷ were not made.

In experiments designed to determine the effects of various in-reactor trap temperatures, the following coolants were used:

<u>Coolant</u>	<u>Theoretical Trap Temperature</u>
Ethanol/ H_2O slush (LN_2)	-30°C to -20°C
n-Octane slush (LN_2)	-56°C
Ethanol/dry ice	-82°C
LN_2	-195°C

In all other runs, the trap was maintained at -82°C using ethanol/dry ice as the coolant.

Experiments involving introduction of additional pressure into the reactor were performed by first achieving stable operation of the reactor at a given flow and output pressure. $\text{N}_2(\text{g})$ was then added to the reactor at an unknown flow rate until a given increase in pressure was obtained. This increase was limited to a few tenths of a torr due to pressure broadening of the ESR spectra at ~ 1.5 torr.

Quantitative Studies - Typical Procedure and Run Conditions

The apparatus was pumped down to a pressure of 15-50 mtorr and all traps and baths filled. The reactor exit valve was closed, the reactor vented, and the H_2O_2 and NaOH solutions (generally 125 ml each) introduced into the reactor. The exit valve was slowly opened and the pressure at the ESR cavity monitored until the background pressure due to H_2O

vaporization and H_2O_2 decomposition decreased below 0.1 torr. At this point the temperature in the reaction mixture had dropped to -10°C to -15°C (depending upon the volume of solution used) due to evaporative cooling. The pH, which had initially been at ~ 9.3 , had increased to ~ 11 due to the effect of the lowered temperature of the solution upon the pH probe. This effect of cooling on the pH was confirmed by similar observations on cooled solutions of ethanol and aqueous NaOH. The Cl_2 flow was brought up to the desired rate and the Cl_2 injector nozzle was lowered into the reaction mixture. This resulted in an increase in the temperature of the reaction mixture to approximately -5°C and a consequent lowering of the pH reading. In approximately 2 minutes, flow and pressure conditions were stable with the cavity pressure between 0.8 and 1.0 torr and the Cl_2 flow rate at 38 - 42 stdcc of air/min. ESR spectra were then recorded.

III. RESULTS

Qualitative Observations

A group of low reactor temperature (-78°C)/concentration experiments yielded no O_2 dimol emission. This was most likely due to low $\text{O}_2(^1\Delta_g)$ production caused by partial or total freezing of the reactants. At higher reactor temperatures it was generally observed that increased flow rate resulted in an increase in dimol emission intensity. This indicated an increased $\text{O}_2(^1\Delta_g)$ production. Similarly, increased system pressure decreased the dimol intensity, implying an increased rate of collisional deactivation of $\text{O}_2(^1\Delta_g)$.

The major utility of the qualitative observations was the indication of general pressure/flow rate regimes in which dimol and iodine emission are probable, Figs. 5 and 6. Dimol emission was not observed at either very low flow rates or system pressures. At large flow rates and system pressures, dimol emission was generally observed. The low pressure threshold for iodine emission appeared to be considerably higher than that for the dimol emission.

Several additional observations included the independence of the dimol and iodine emissions. It was possible to obtain either emission with or without the presence of the other. Both types of emission tended to cease abruptly, generally after ~10 minutes of reactor operation. This last observation may indicate a threshold reactant concentration or pH for $O_2(^1\Delta_g)$ production or alternately, a minimum $O_2(^1\Delta_g)$ pressure for dimol and/or iodine emission.

Quantitative Results

Standardization

Although the standardization of the doubly integrated ESR spectra with $O_2(^3\Sigma_g)$ pressure is straightforward, a simple error was observed in several early results. The plot of the double integral versus pressure should be linear, whereas several standardization curves showed a distinct non-linearity at large pressure, Fig. 7. This decrease in the double integral magnitude was observed to be caused by the relatively narrow ESR magnetic sweep range. For large pressure, narrow sweeps tended to disregard too much of the spectral wings, resulting in a decreased double integral. When a larger magnetic field sweep width was used at higher pressures,

the double integral versus pressure plots became linear. This observation indicates that in taking ESR spectra over various pressures, care should always be exercised to guarantee that no significant signal is lost in the spectral wings.

The slope and intercept of a linear least squares fit to the data in Fig. 4 were found to be,

$$DI = 3.5664P + 0.0623 \quad (2)$$

where DI is the double integral of the ESR spectrum and P is the O_2 pressure.

Standard Run

A preliminary experiment to observe the general trends of the chemical generator was performed. Plots of the various parameters observed and the pressures and percentages generated from the ESR spectra are given in Figs. 8a - f. A slow increase in reactor temperature was observed throughout the experiment. It is unclear if the corresponding decrease in pH during the reaction is an accurate observation or a result of operational uncertainties of the pH probe at the temperatures used. Separate pH readings of standard solutions showed anomalous behavior at temperatures at and below -5°C . The pressure versus time plot illustrates an initial increase in the $O_2(^3\Sigma_g^-)$ pressure and a very slight decrease in $O_2(^1\Delta_g)$ pressure over the entire reaction. These results are further accentuated by the percent oxygen versus time plots which show a steady total percentage of O_2 and decreasing $O_2(^1\Delta_g)$ percentage after an initial increase in both values. The majority of the gas flow leaving the chemical reactor is O_2 , indicating near complete reaction of the $Cl_2(g)$ at the flow rate of

40 stdcc of air/min. This is supported by the lack of visible amounts of Cl_2 condensed on the liquid nitrogen cold trap positioned downstream from the ESR cavity. The drastic variation in all parameter plots after 160 min is due to an increase in the temperature of the in-reactor cold trap which was allowed to warm. This result will be discussed below.

Cl_2 Flow Rate

Variations of Cl_2 flow rate was observed to significantly affect the production of O_2 . Increasing Cl_2 flow increased the O_2 pressure slightly but decreased the % O_2 in the total gas flow. This might possibly be due to inclusion of unreacted Cl_2 in the reactor output. A definite dependence of reactor temperature upon the Cl_2 flow rate was observed. Increased flow rate caused an increase in the reactor temperature. In Figs. 9a - f are shown the effects of decreasing the Cl_2 flow rate by approximately 50%. The data points at 52, 54.5, 73.5 and 77 minutes are at reduced Cl_2 flows. In agreement with the previous statement, the reactor temperature decreases for decreased Cl_2 flow rates. The pH data is suspect due to problems with the probe mentioned earlier. The pressures of $\text{O}_2(^3\Sigma_g)$ and $\text{O}_2(^1\Delta_g)$ both decrease whereas the percentages of O_2 in the gaseous product are essentially unaffected by the decreased Cl_2 flow.

Trap Temperature

Several early experiments indicated a dependence of the O_2 ESR signal upon the temperature of the in-reactor cold trap. An increase in trap temperature appeared to increase the pressure and percentage of $\text{O}_2(^1\Delta_g)$ (last five points of Figs. 8e and f). Further warming above a trap temperature of approximately -25°C caused a drop in $\text{O}_2(^1\Delta_g)$ pressure and

percentages. In Figs. 10a - g, results using a series of different trap temperatures are presented. The first (<20 min) and last (>100 min) groups of points are for a standard EtOH/CO₂(s) trap which has a temperature of approximately -78°C. An EtOH/H₂O(s) trap mixture maintains a temperature of approximately -30°C. At this temperature a significant decrease in O₂(³Σ_g) pressure and total % O₂ was observed. The O₂(¹Δ_g) pressure remains fairly constant at this temperature which results in an increase in % O₂(¹Δ_g). For a trap temperature of approximately -45°C, the O₂(³Σ_g) pressure and total % O₂ return to values expected for the standard trap temperature of -78°C. When the trap temperature is decreased significantly, -196°C, the O₂(¹Δ_g) signal is completely quenched. This is possibly due to increased residence times and consequent deactivation of the O₂(¹Δ_g) on the trap surface.

Nozzle Position and N₂ Agitation

A preliminary investigation of reactor nozzle position suggested that the O₂ product ratio depended upon the position of the Cl₂ injection nozzle relative to the fluid reactant/gas interface (Fig. 3). The results of a further study of this effect are presented in Figs. 11a - f. An insensitive regulator valve resulted in considerable drift in the Cl₂ flow rate for this experiment. This makes interpretation of the results difficult. Problems with the ESR spectrometer involved additional uncertainty for several points (101.5 to 122 min). For t < 40 min the standard reactor configuration was used. For 40 < t < 70 min a small flow of N₂ was bubbled through the reactant solution causing splashing of the reactants onto the reactor walls. A slight decrease in both O₂(³Σ_g) and O₂(¹Δ_g)

pressures was observed. Although the total % O_2 decreased, due to N_2 in the gas flow, the % $O_2(^1\Delta_g)$ remained the same as for the standard conditions. For the points at 74 and 76 min, the Cl_2 nozzle was raised out of the reactants to its maximum position. Both $O_2(^3\Sigma_g)$ and $O_2(^1\Delta_g)$ pressures decreased but the % $O_2(^1\Delta_g)$ increased under these conditions. The decrease in total O_2 was evidently due to unreacted Cl_2 flowing out of the reactor chamber, as indicated by considerable Cl_2 formation on the $N_2(l)$ trap. For the points at 81.5, 84.5, and 87 min the Cl_2 nozzle was lowered to 1 cm above the liquid reactant level. This decreased slightly the Cl_2 excess as indicated by the slight increase in total % O_2 and increased the mean % $O_2(^1\Delta_g)$. For the points at 91.5, 96, and 99 min the N_2 flow was stopped while maintaining the Cl_2 nozzle above the liquid interface. This caused a drop in the total % O_2 to near zero, as the reaction was essentially halted. The points at $t > 122$ min are for the standard reactor configuration. The results agree well with what would be expected for standard conditions.

Reactant Volumes

Two experiments were run with other than the standard 125 ml of both reactants. For 85 ml of both reactants, the % $O_2(^1\Delta_g)$ was observed to be unaffected. The total % O_2 was slightly decreased for the smaller volume, compared to the standard volume. This possibly indicates incomplete reaction of the Cl_2 with the liquid reactants. The second experiment used 250 ml of 6M NaOH and 170 ml of 90% H_2O_2 . A comparison of these results with the standard volumes indicates a decreased O_2 production. $O_2(^1\Delta_g)$ pressure, $O_2(^3\Sigma_g)$ pressure, % $O_2(^1\Delta_g)$ and total % O_2 all decrease for the unequal volume experiment.

IV. CONCLUSIONS

It has been found that the present chemical generator is an easily handled physical configuration which generates reproducible quantities of $O_2(^1\Delta_g)$. Although several minor variations need to be made, the present generator should be satisfactory for most of the physical optimization and mechanistic experiments run in the immediate future. Several general conclusions which can be drawn from the present work are briefly tabulated below.

(1) From the ESR standardization experiments it is evident that care must be exercised to guarantee that no significant ESR signal is lost to the unrecorded spectral wings. This requires choice of appropriate magnetic field sweep widths for given O_2 pressures.

(2) The standard reaction conditions indicate nearly complete reaction of the $Cl_2(g)$. By increasing the Cl_2 flow rate or decreasing reactant volume, unreacted Cl_2 can be observed downstream from the ESR cavity. This implies an optimum Cl_2 flow rate exists, for a given reactant volume, for O_2 production and minimization of unreacted Cl_2 .

(3) The in-reactor cold trap temperature variation indicates an optimum temperature for % $O_2(^1\Delta_g)$ between $-20^\circ C$ and $-35^\circ C$. Liquid nitrogen trap temperatures result in the loss of the $O_2(^1\Delta_g)$ ESR signal.

(4) The nozzle position and agitation studies indicate a definite surface effect upon $O_2(^1\Delta_g)$ production. To optimize the % $O_2(^1\Delta_g)$ it is necessary to decrease bulk fluid diffusion of the $Cl_2(g)$ while increasing the Cl_2 /liquid reactant interface. This process not only increases the % $O_2(^1\Delta_g)$ but might economize on the usage of $Cl_2(g)$.

(5) It is apparent from the present results that a better electrode must be found for pH measurements in the reactant solution. Both the solution temperature and the general corrosive nature of the reactants makes the present glass electrode unsatisfactory.

Several future investigations are evident from the present work. Several of these are tabulated below.

(1) Further trap temperature studies, involving better control of the trap temperature, need to be performed to determine the optimum value for the present chemical generator.

(2) Further reactant volume and concentration analysis should be carried out, possibly in conjunction with Cl_2 flow rate considerations.

(3) The nozzle position and N_2 agitation study needs to be repeated with better control of the Cl_2 flow rate.

(4) A better pH monitoring device needs to be found and used routinely in these experiments.

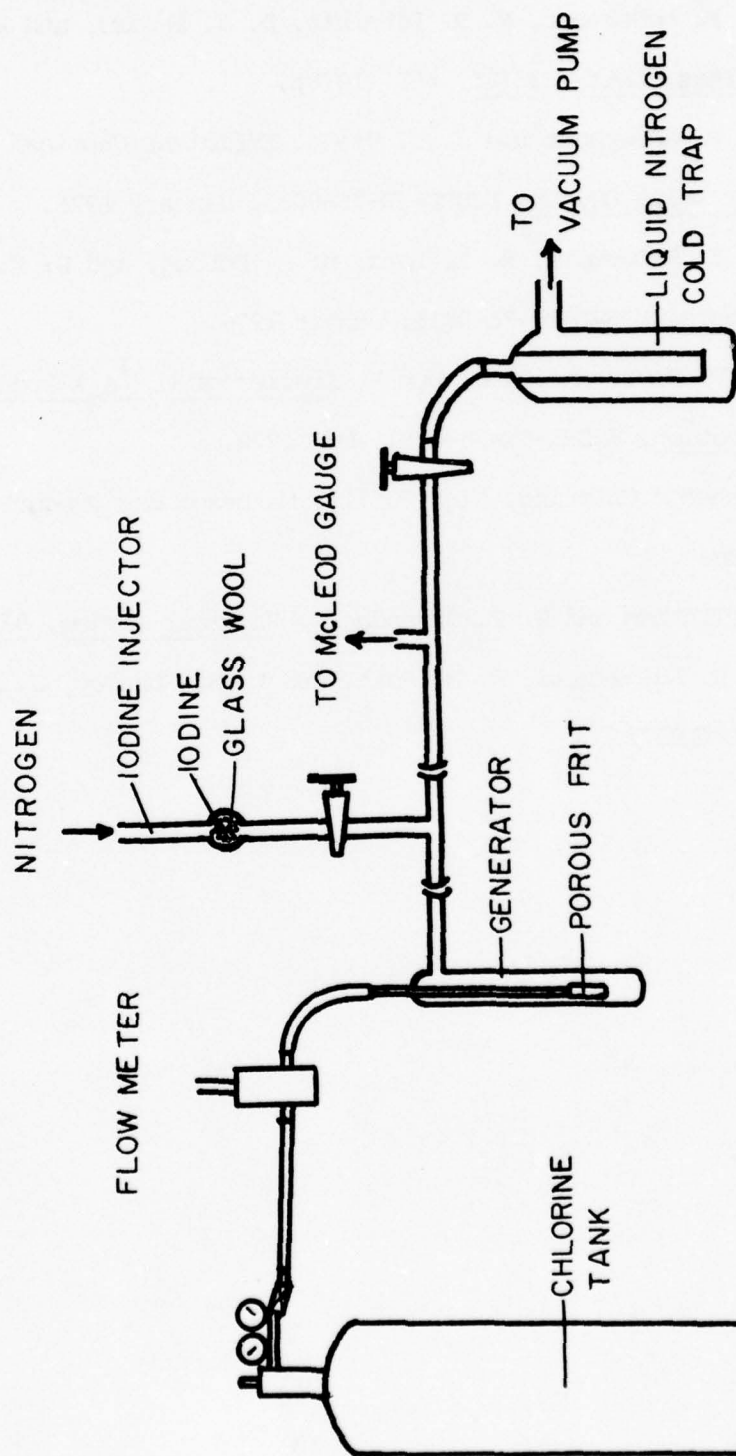
(5) An attempt to optimize $\text{Cl}_2(\text{g})$ /reactant surface interface should be made.

(6) Control and variation of the reactant solution temperature is presently being considered.

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FIG. 1



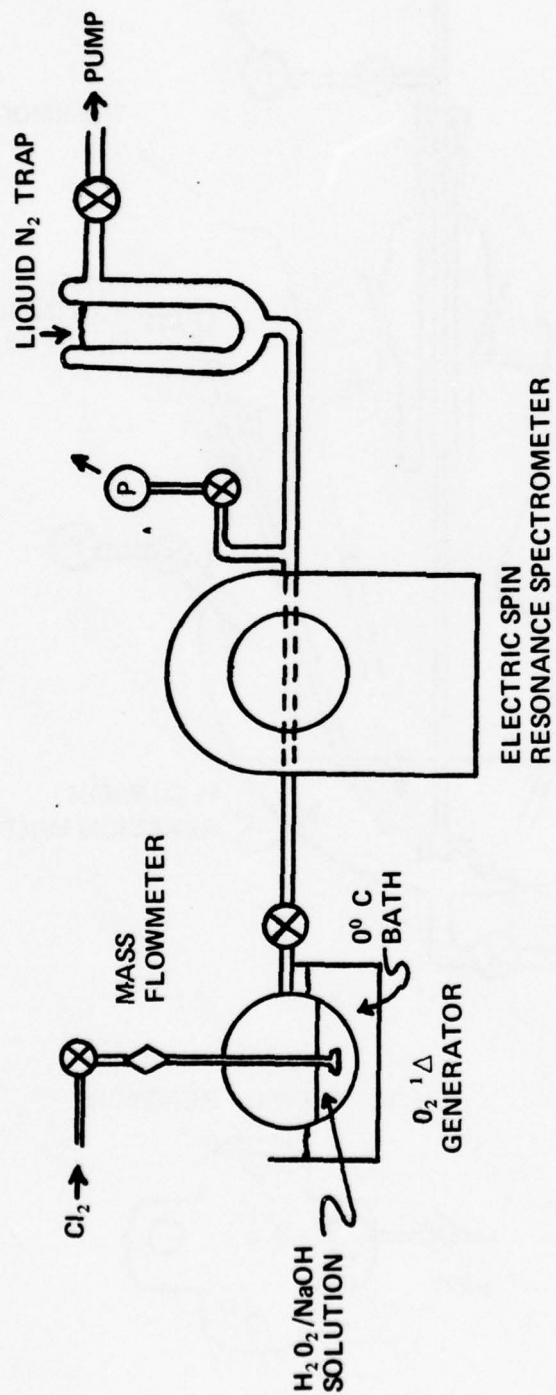
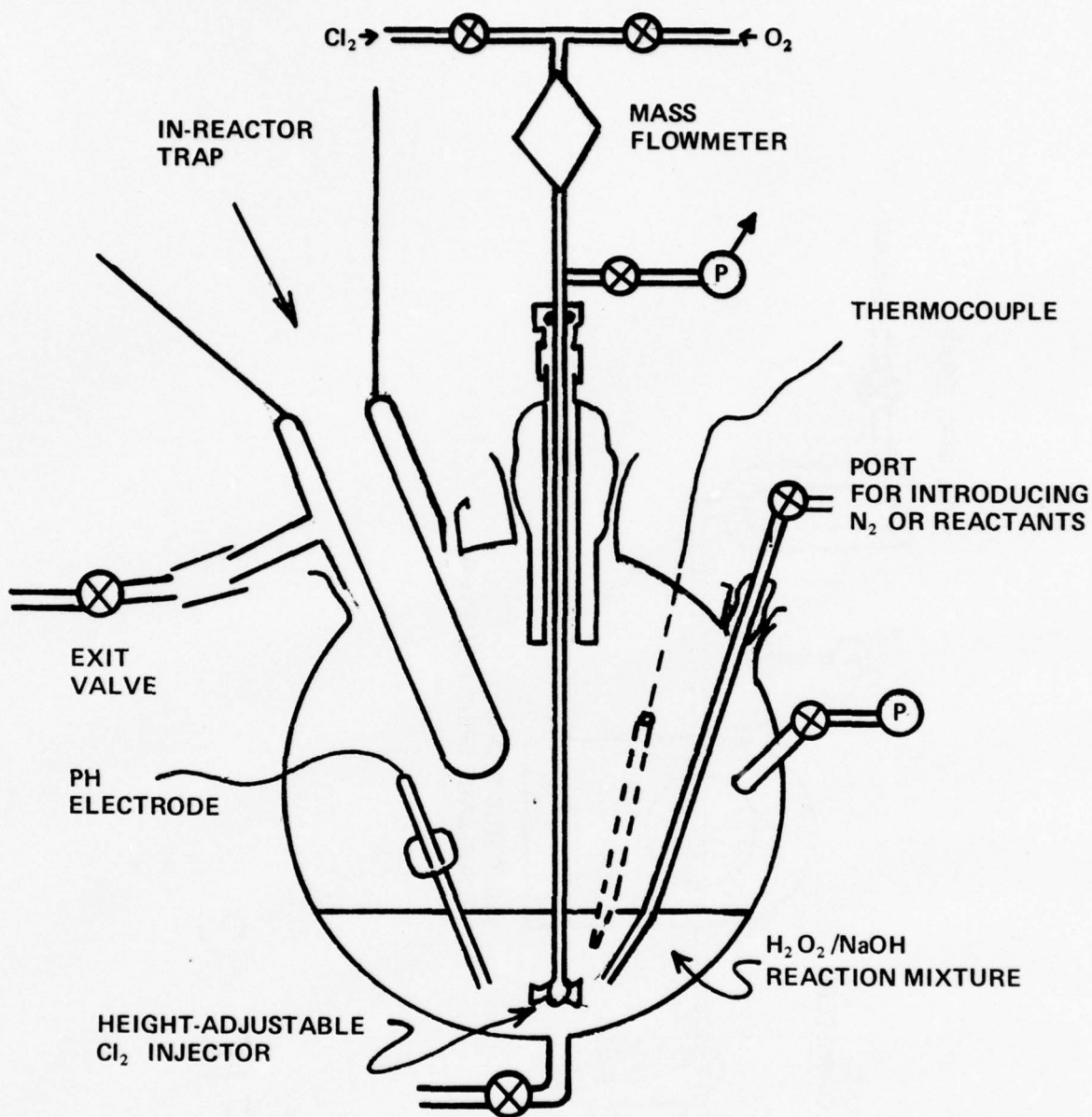


FIG. 2



TOP VIEW OF REACTOR

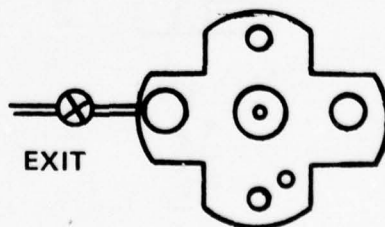


FIG. 3

FIG. 4

CALIBRATION CURVE

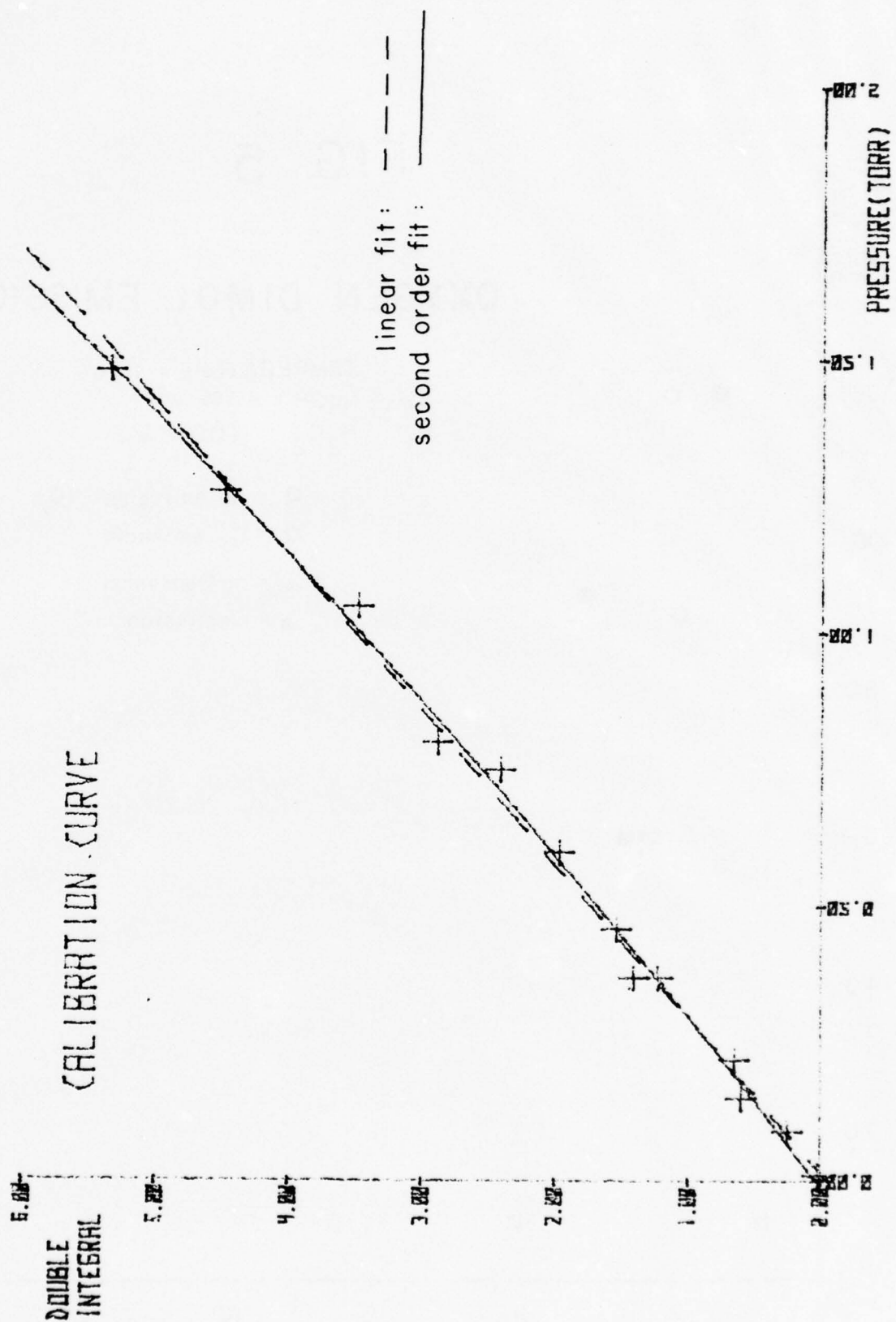


FIG. 5

OXYGEN DIMOL EMISSION

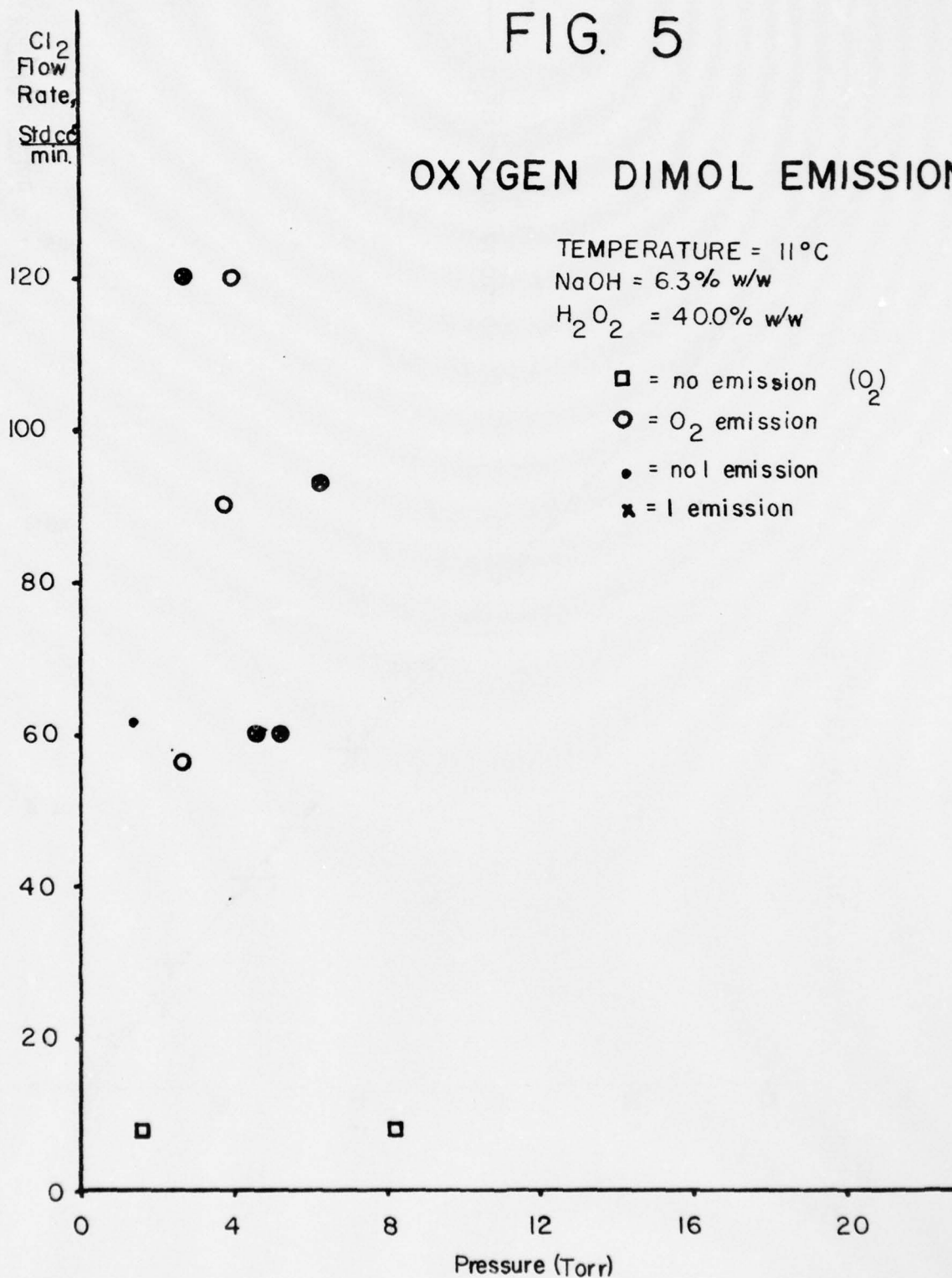


FIG. 6

OXYGEN DIMOL EMISSION

Cl₂
Flow
Rate
(Stdcc/min)

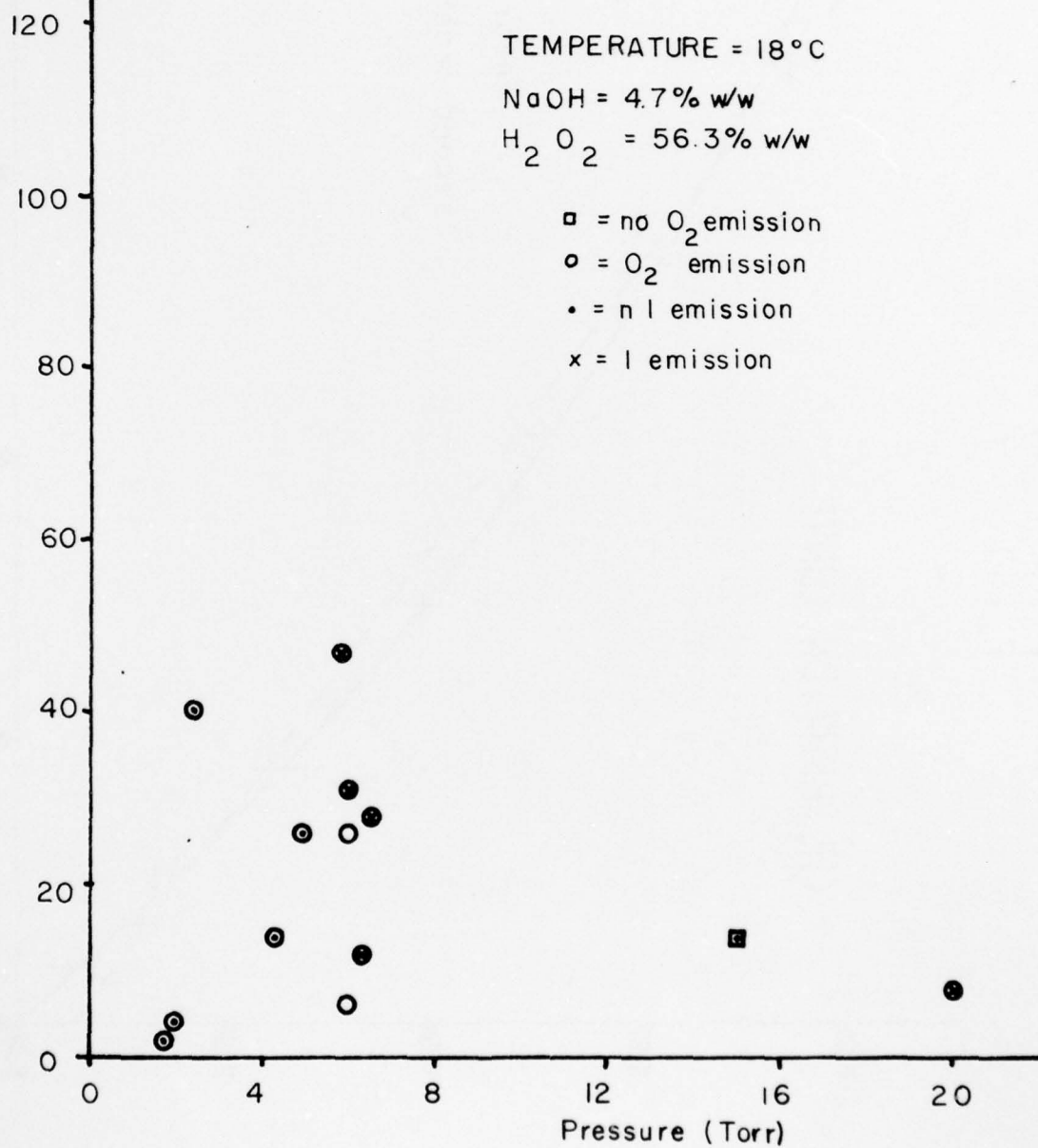


FIG. 7

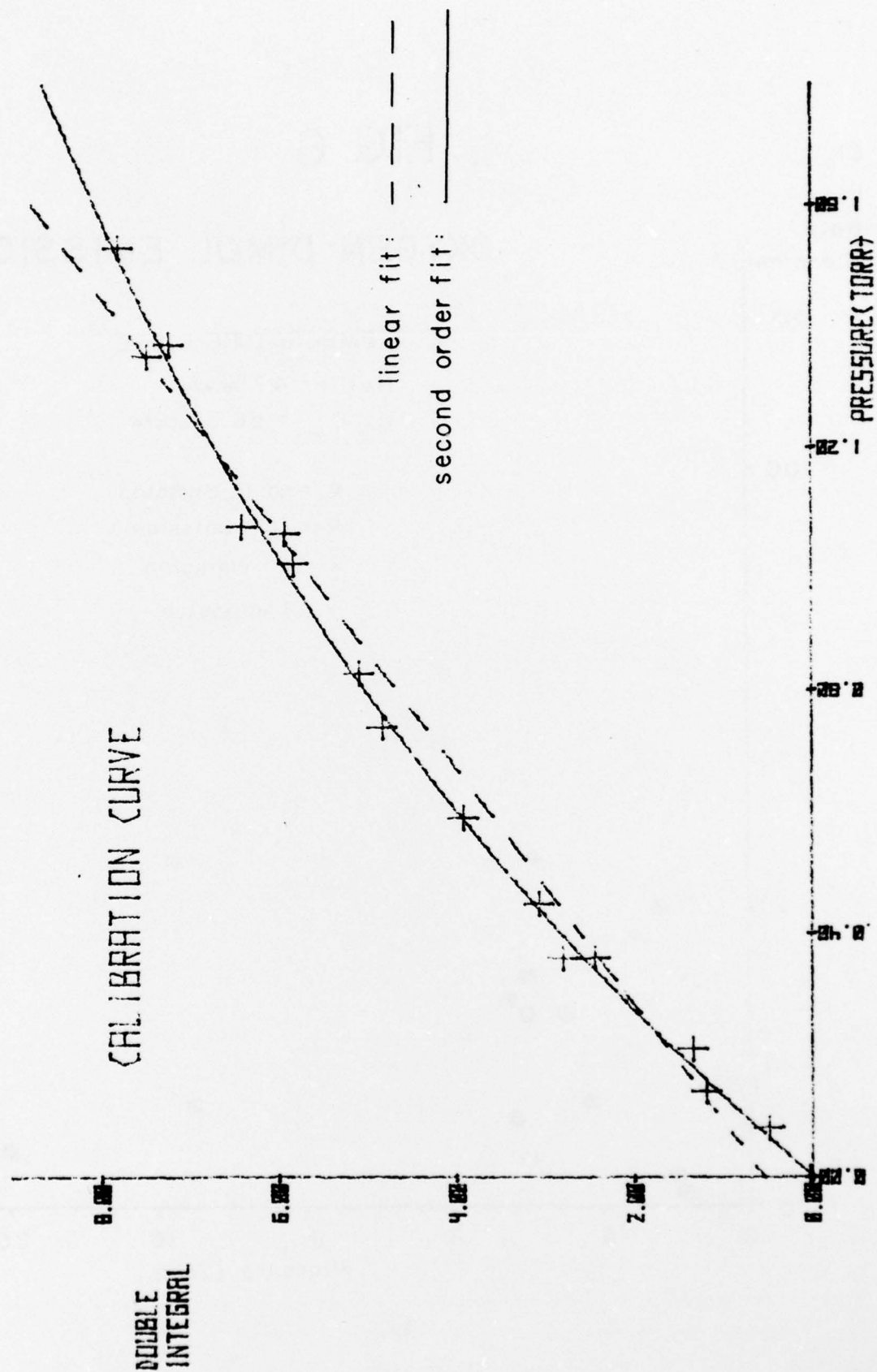


FIG. 8

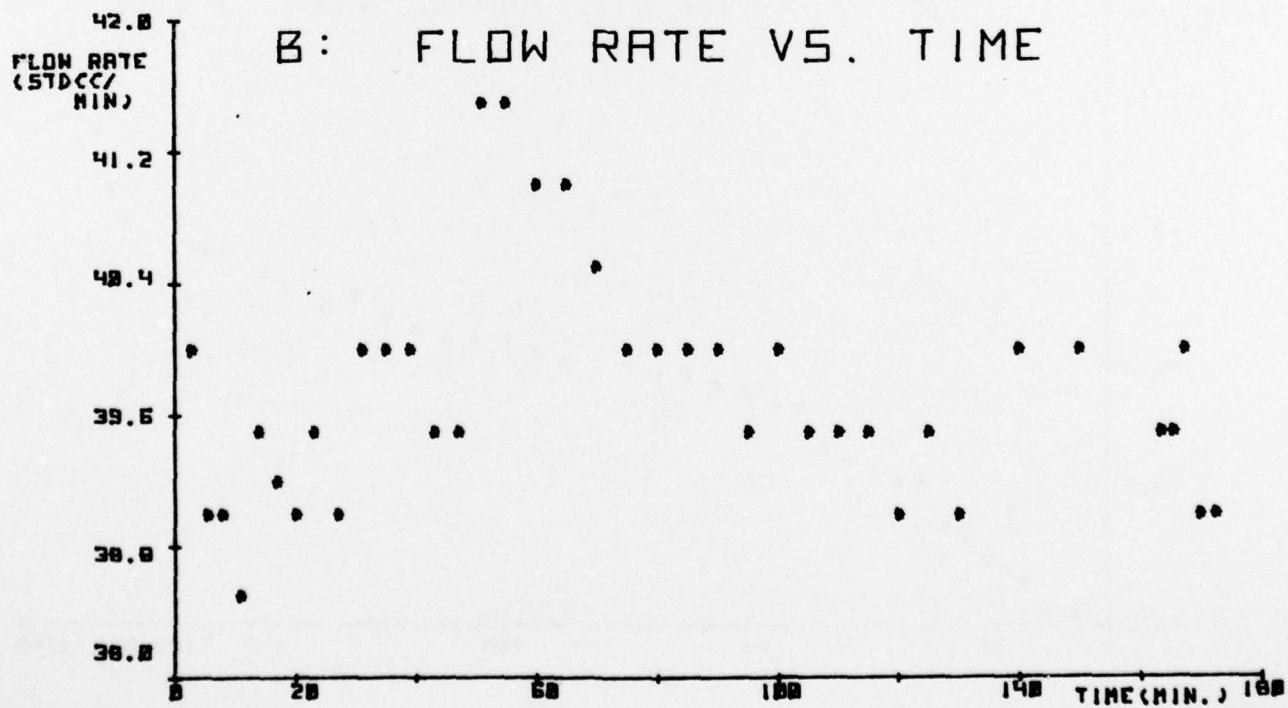
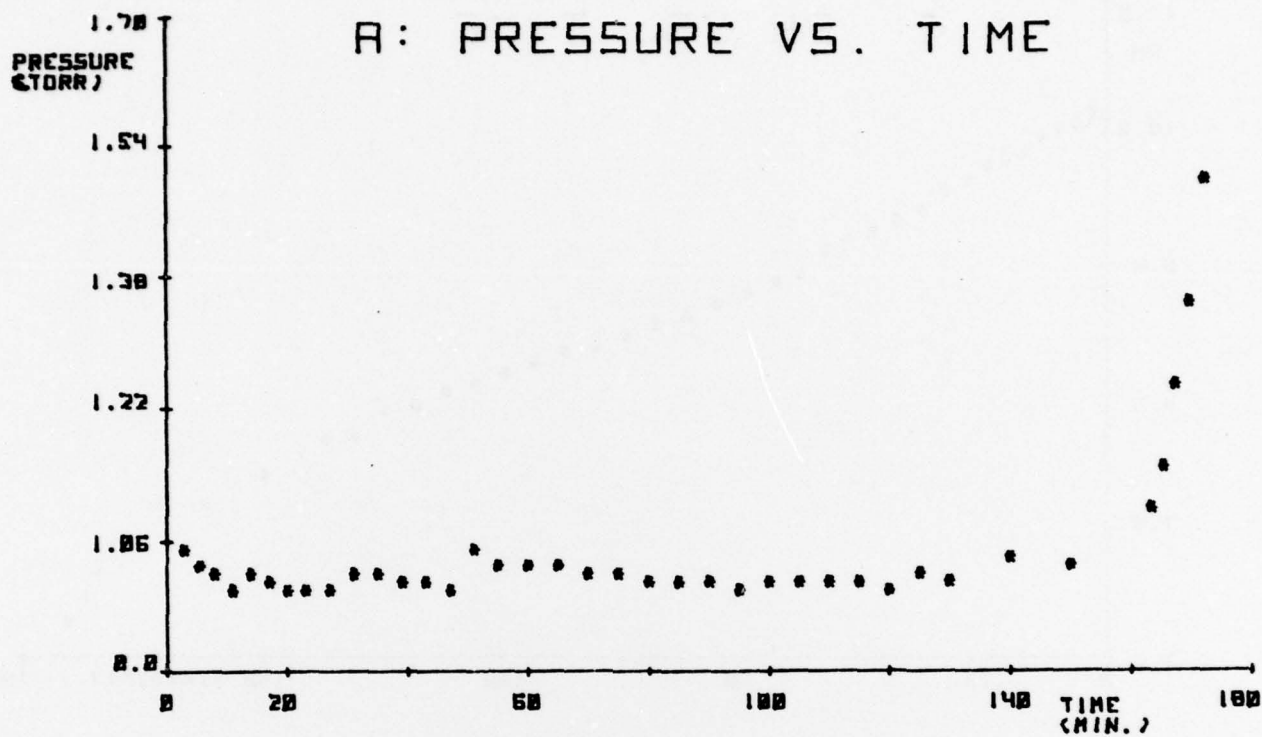


FIG. 8

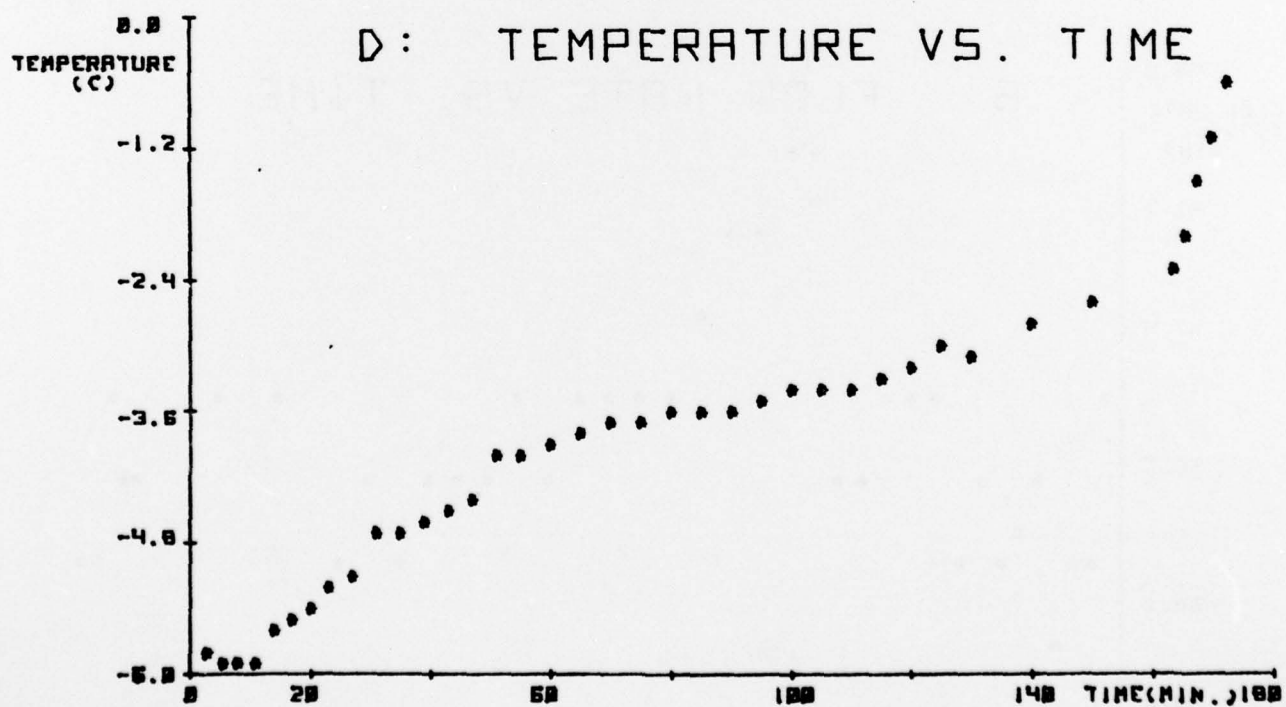
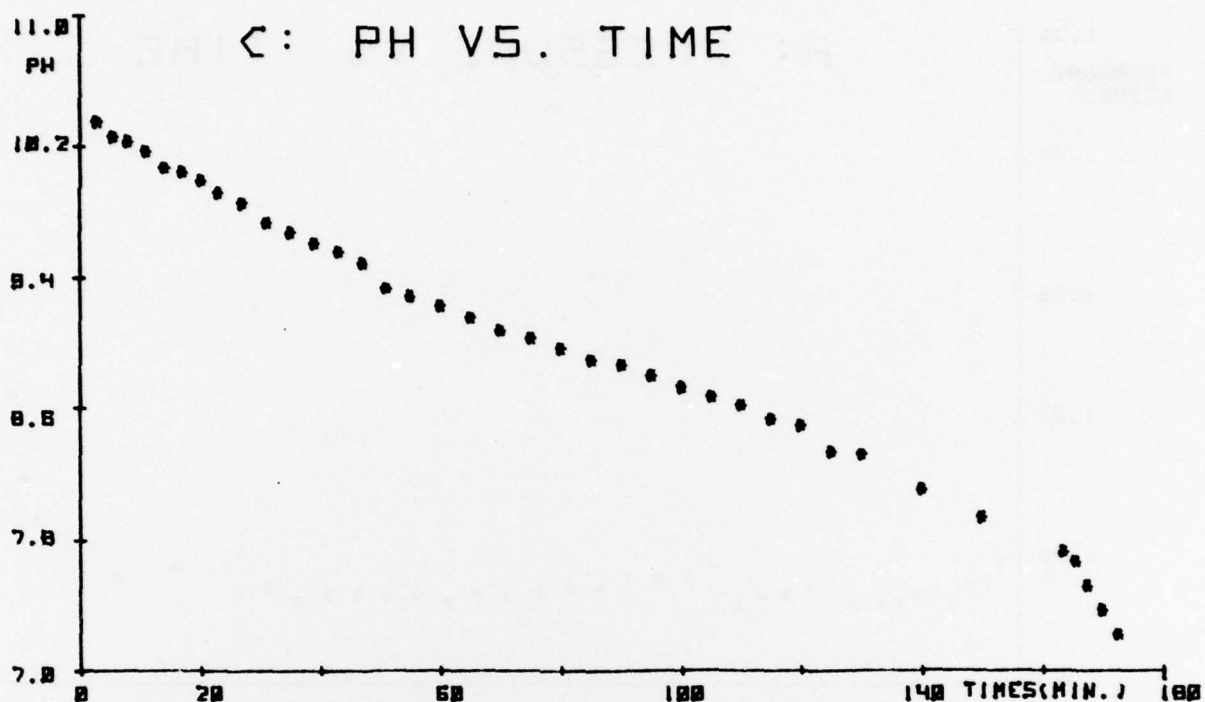


FIG. 8

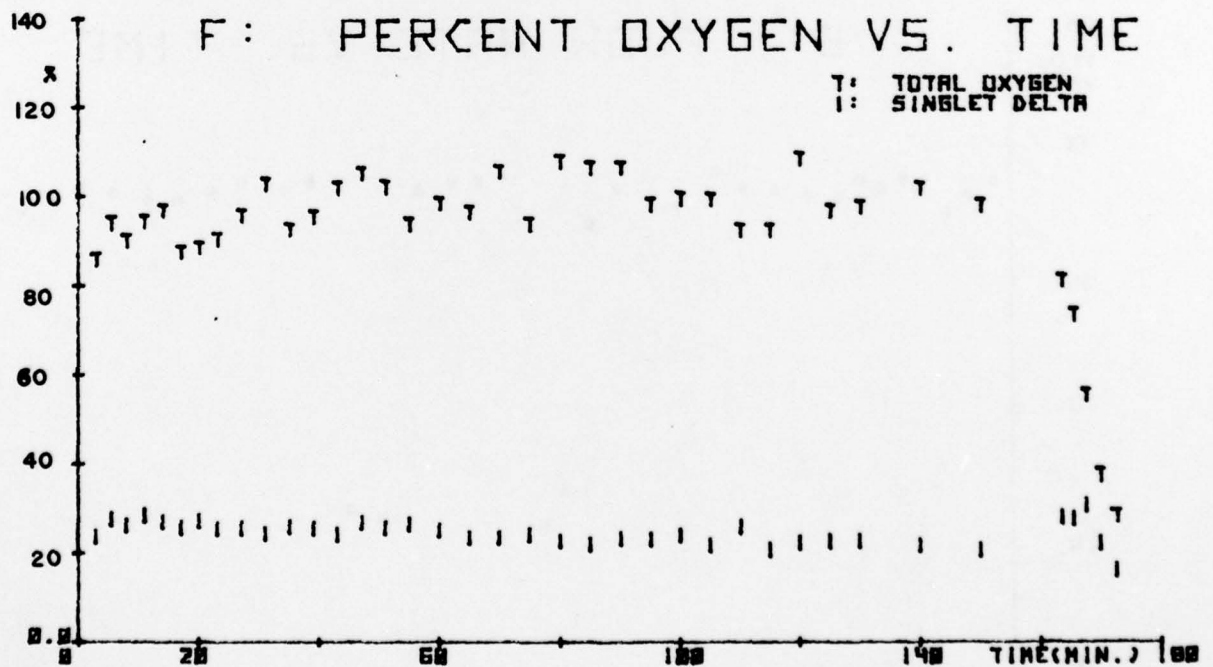
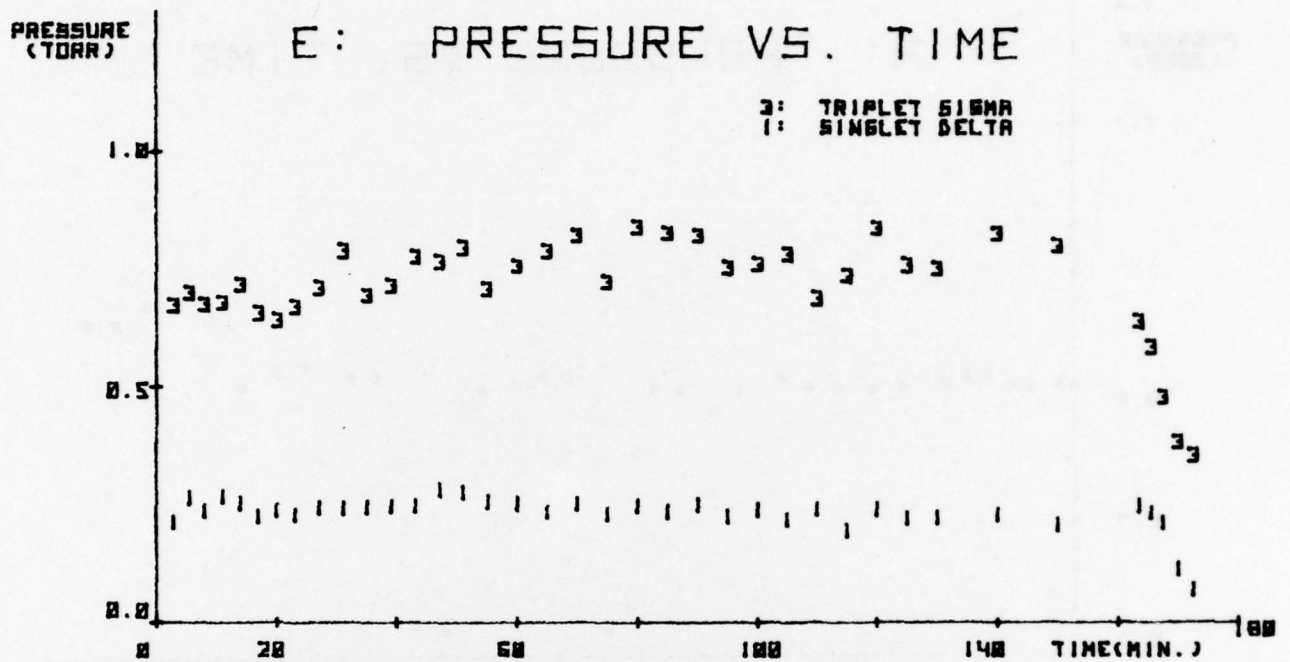


FIG. 9

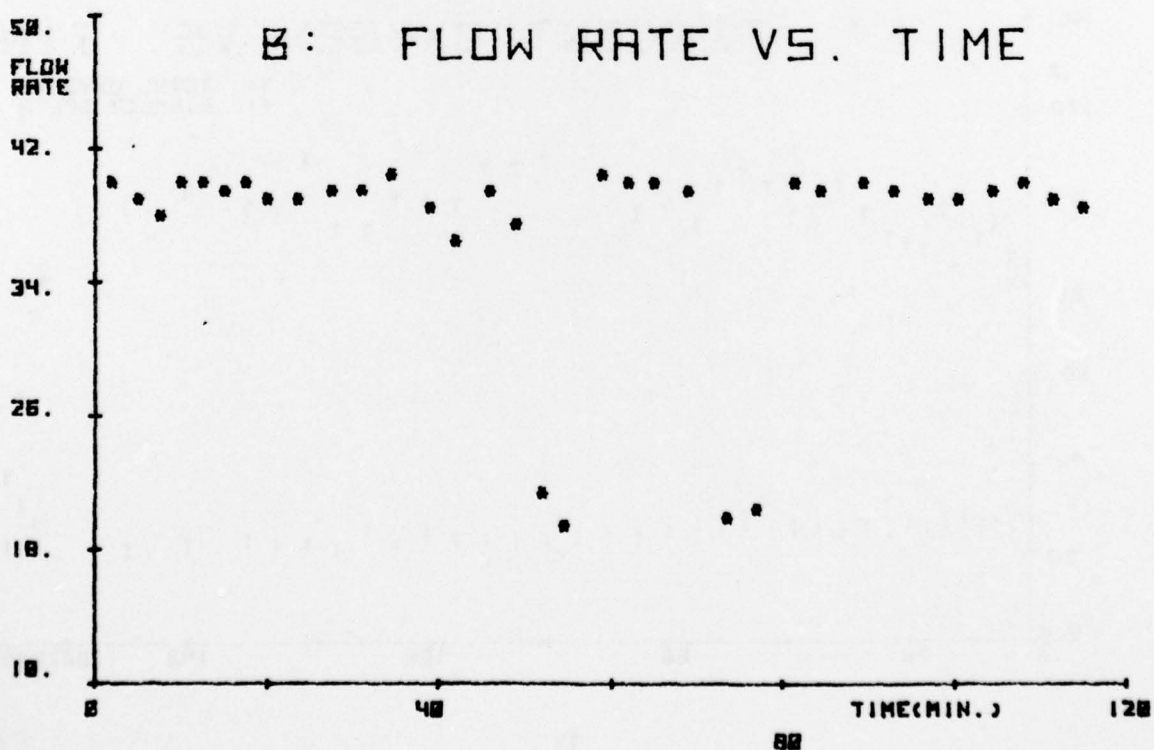
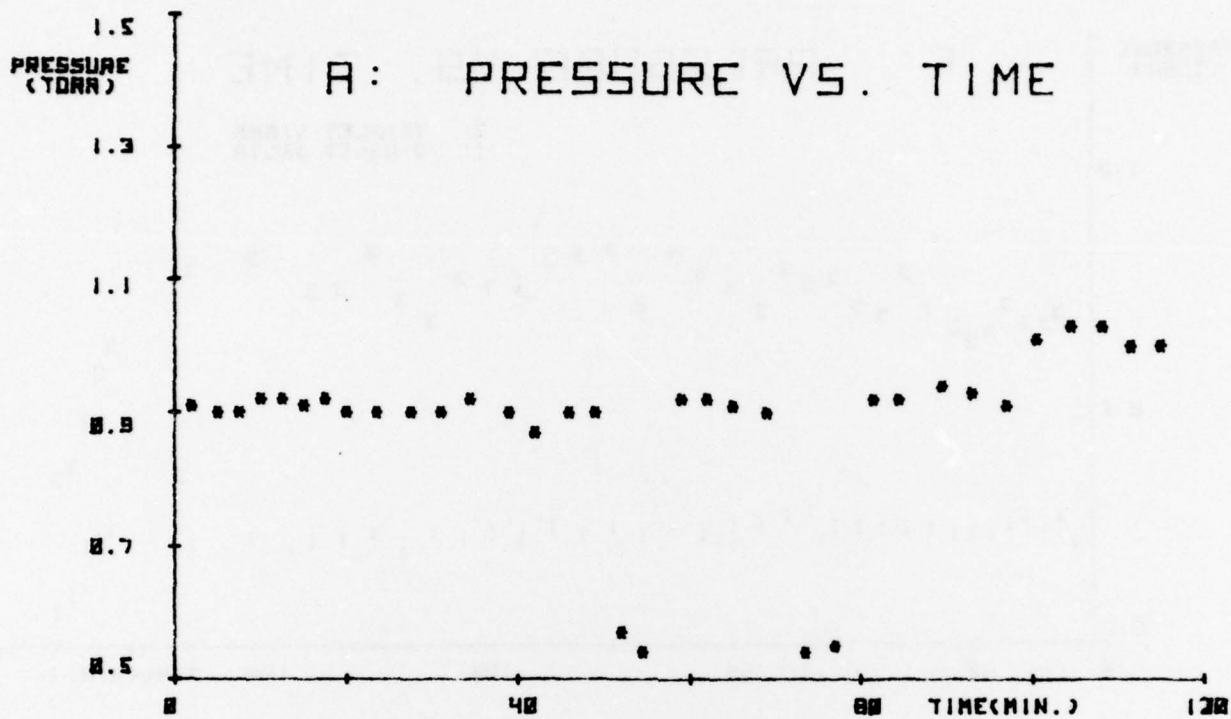


FIG. 9

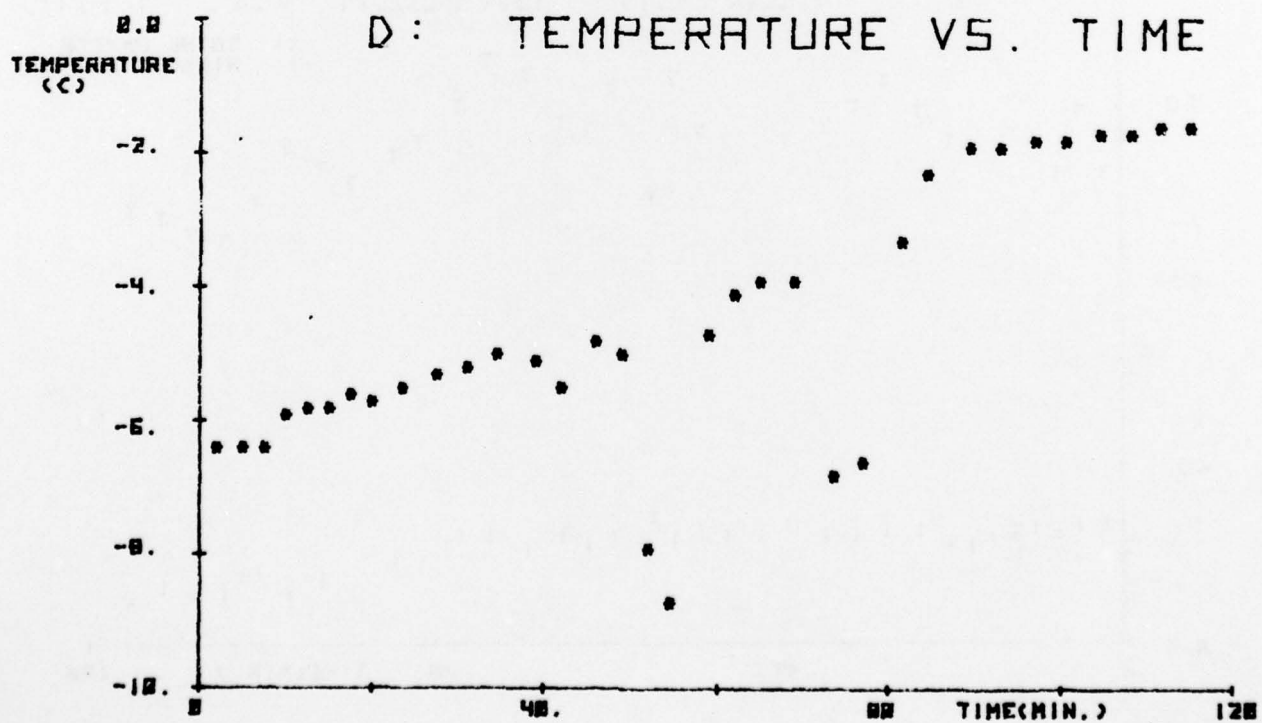
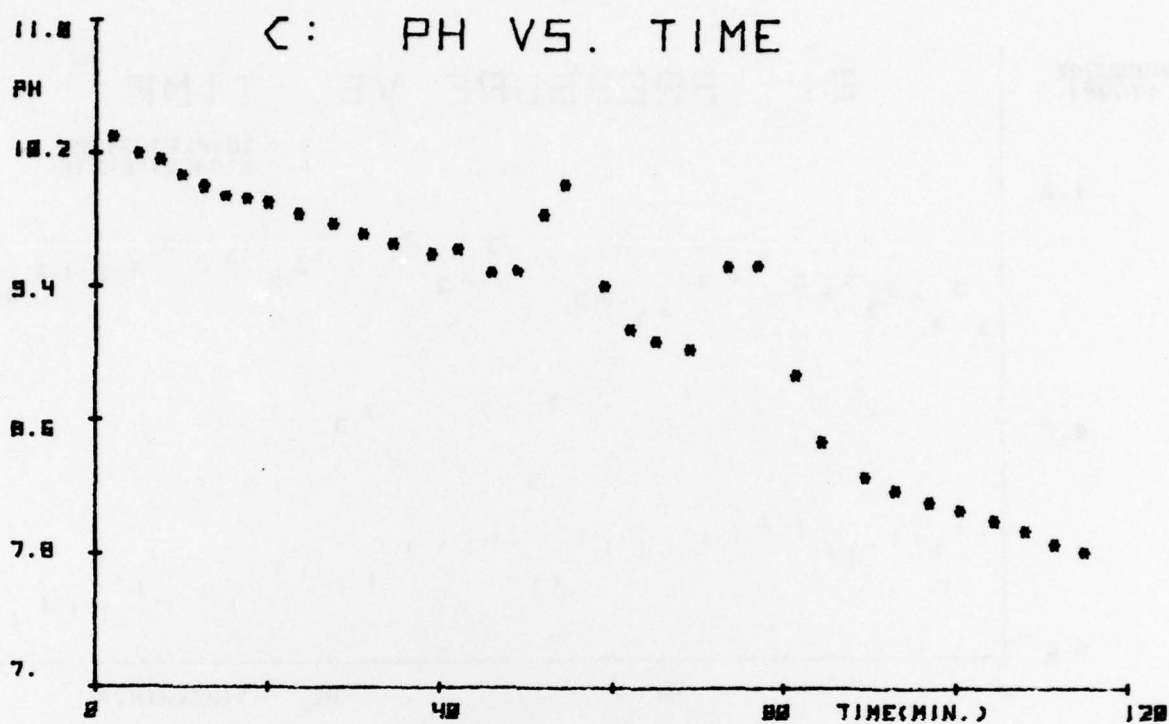


FIG. 9

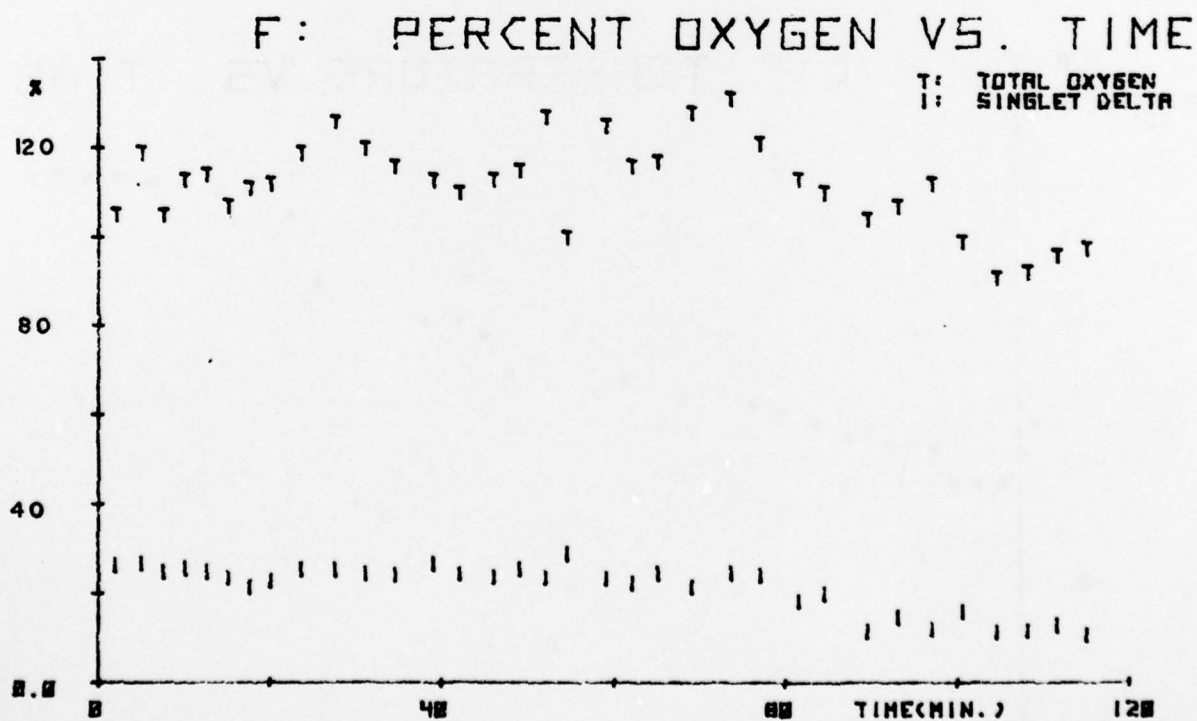
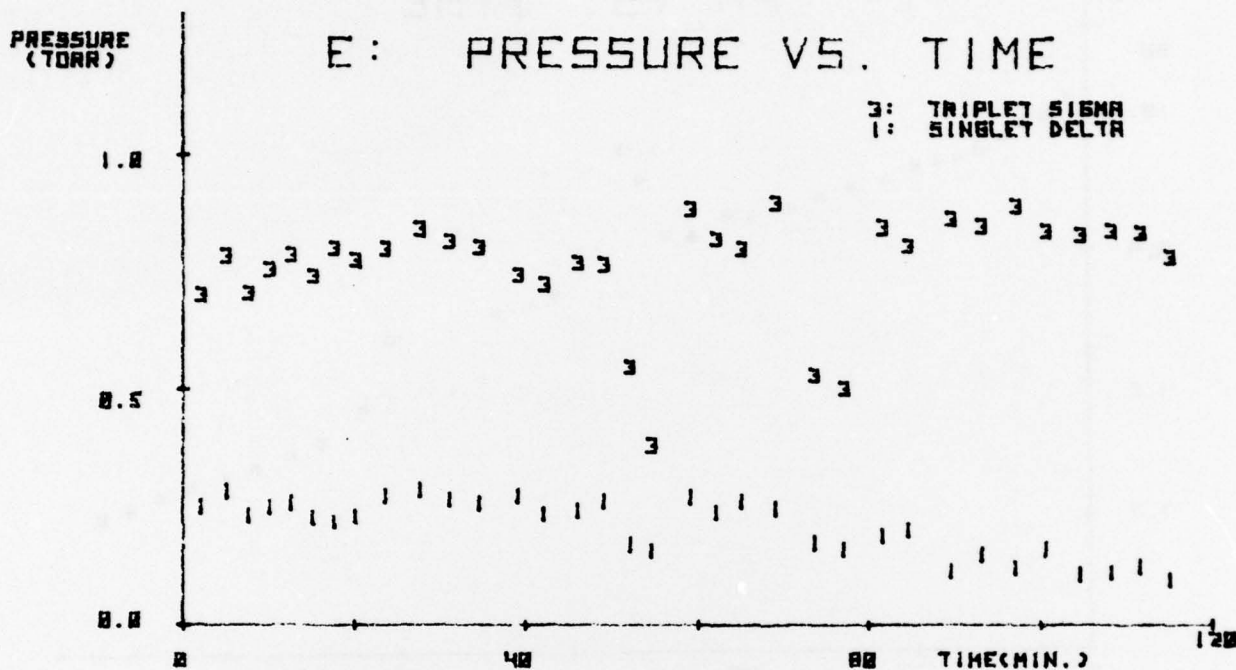


FIG. 10

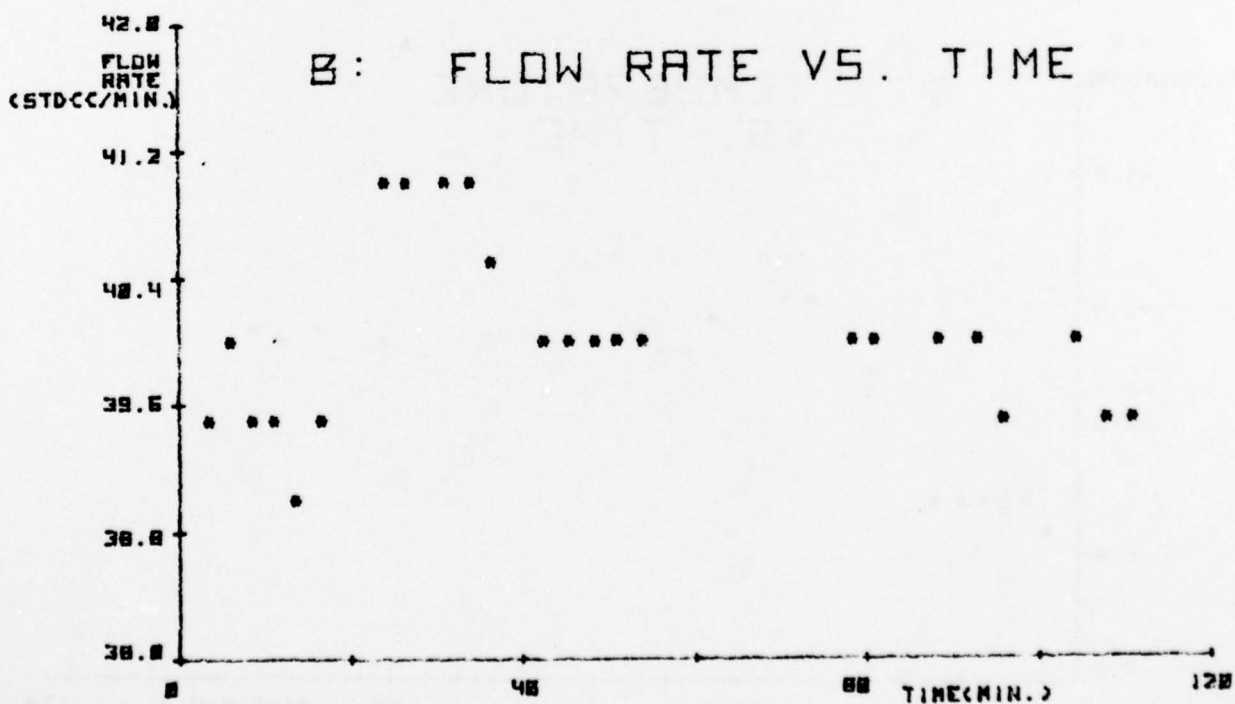
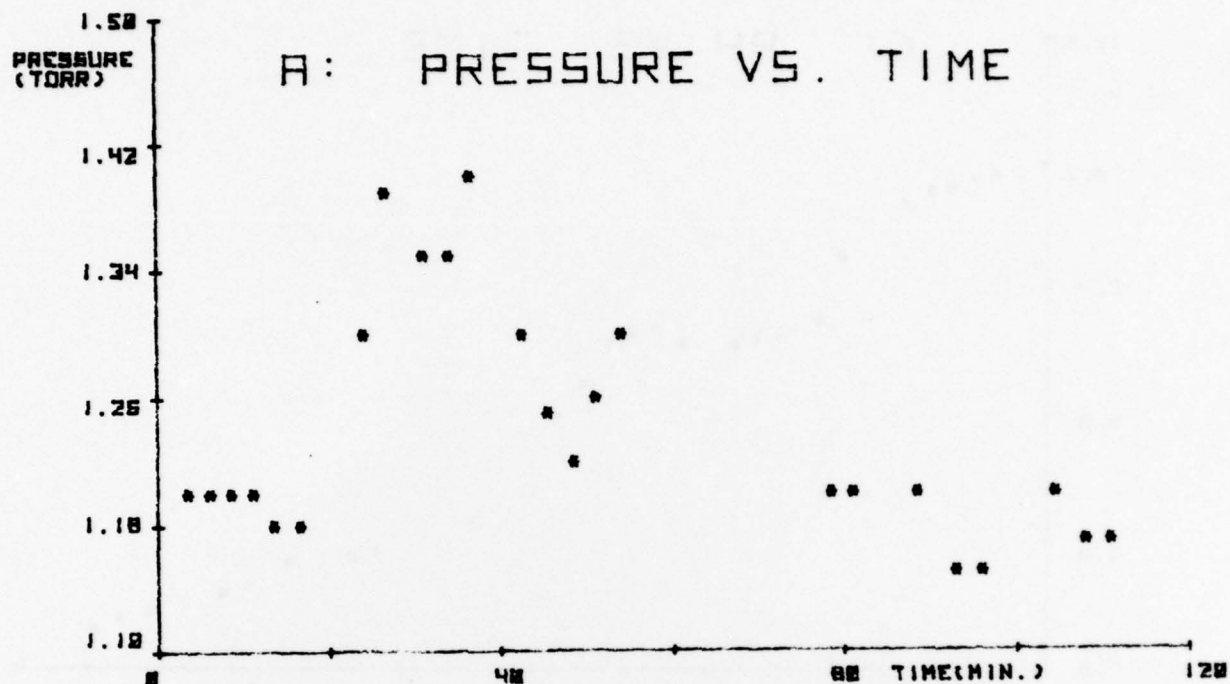


FIG. 10

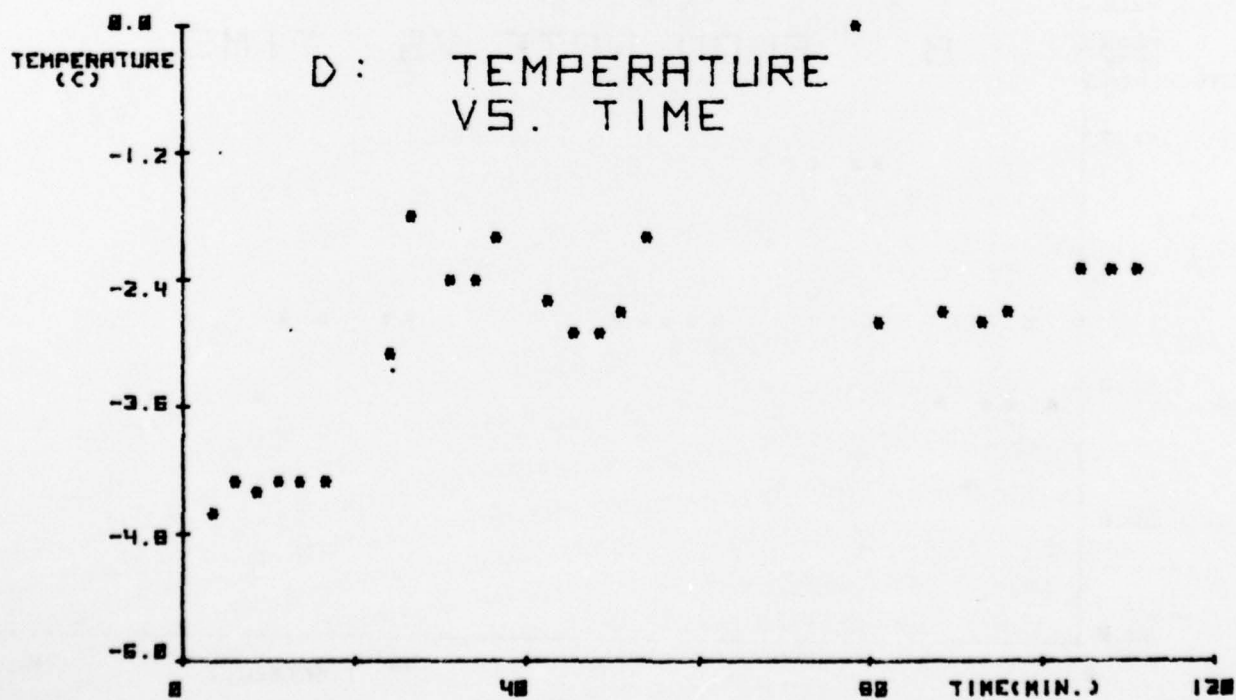
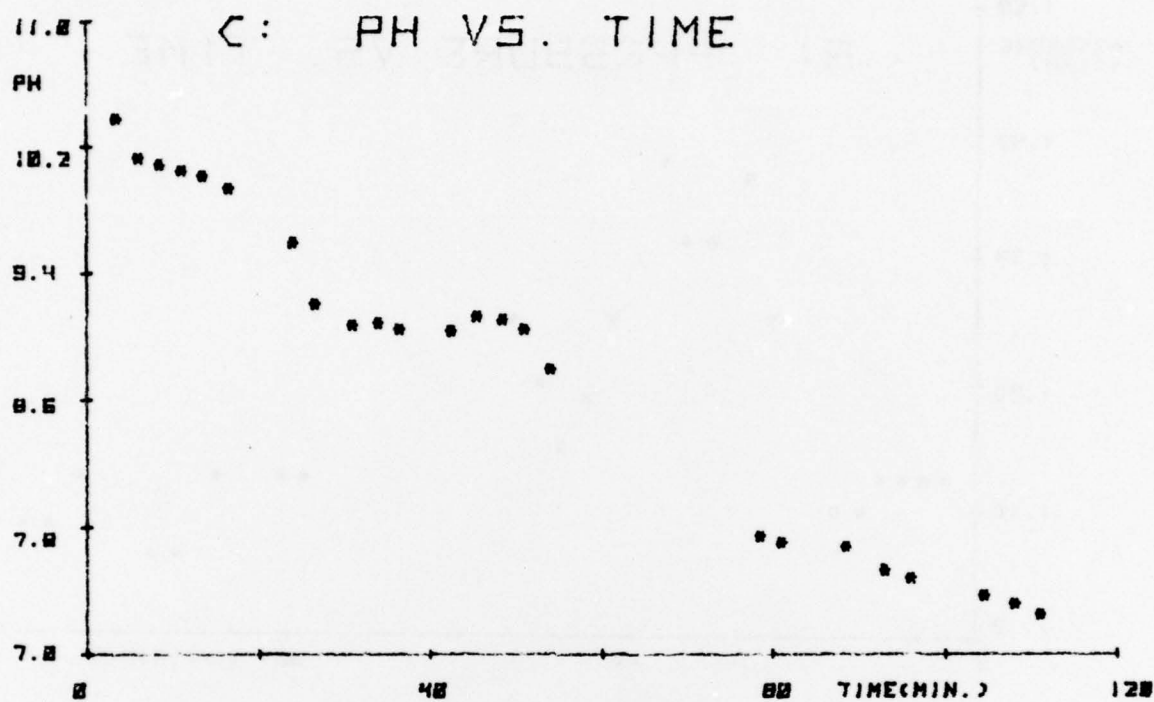


FIG. 10

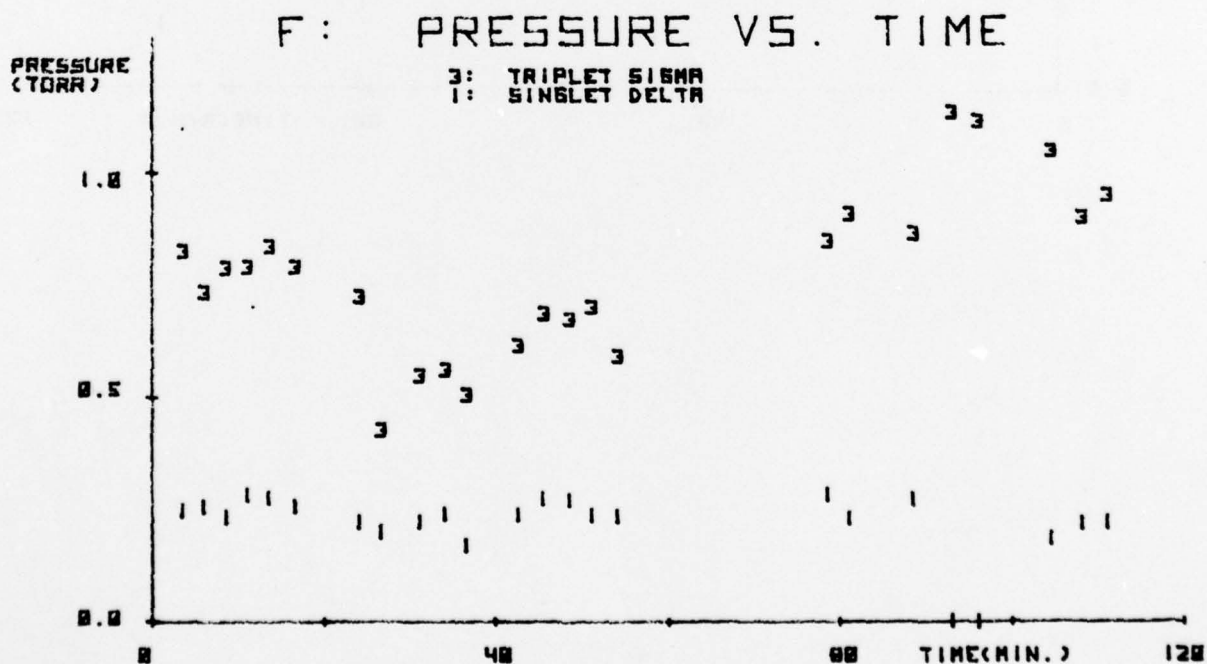
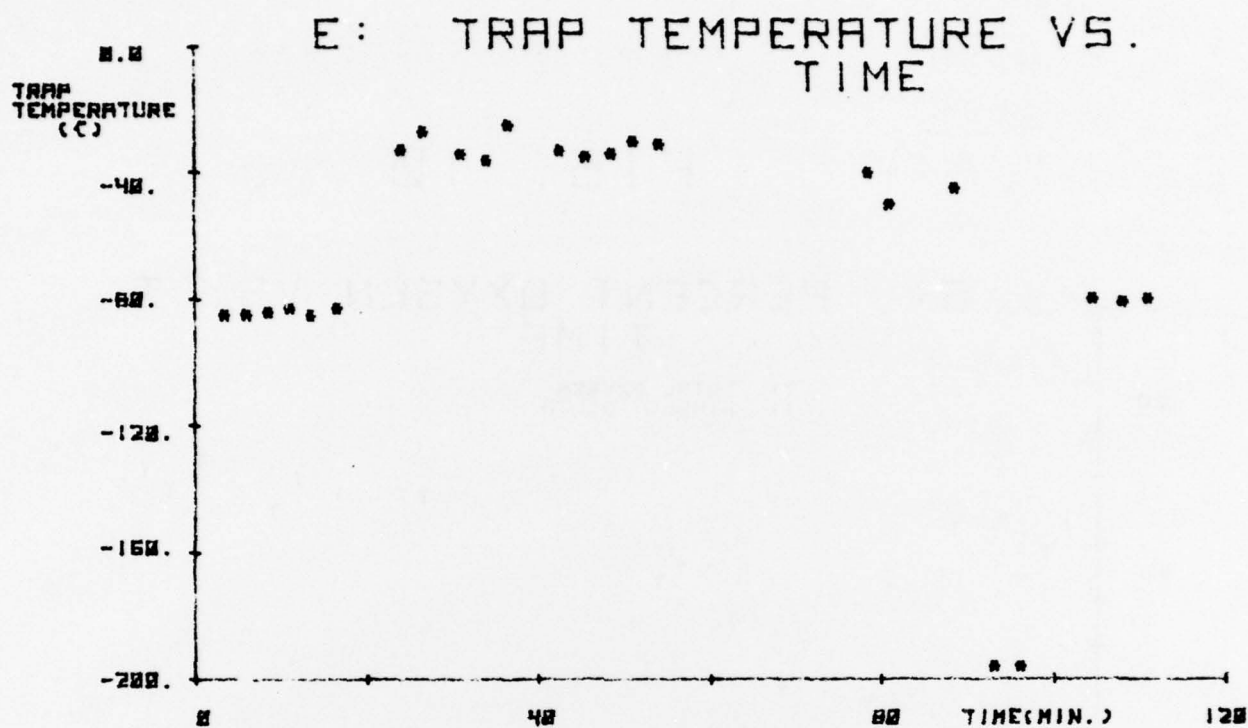


FIG. 10

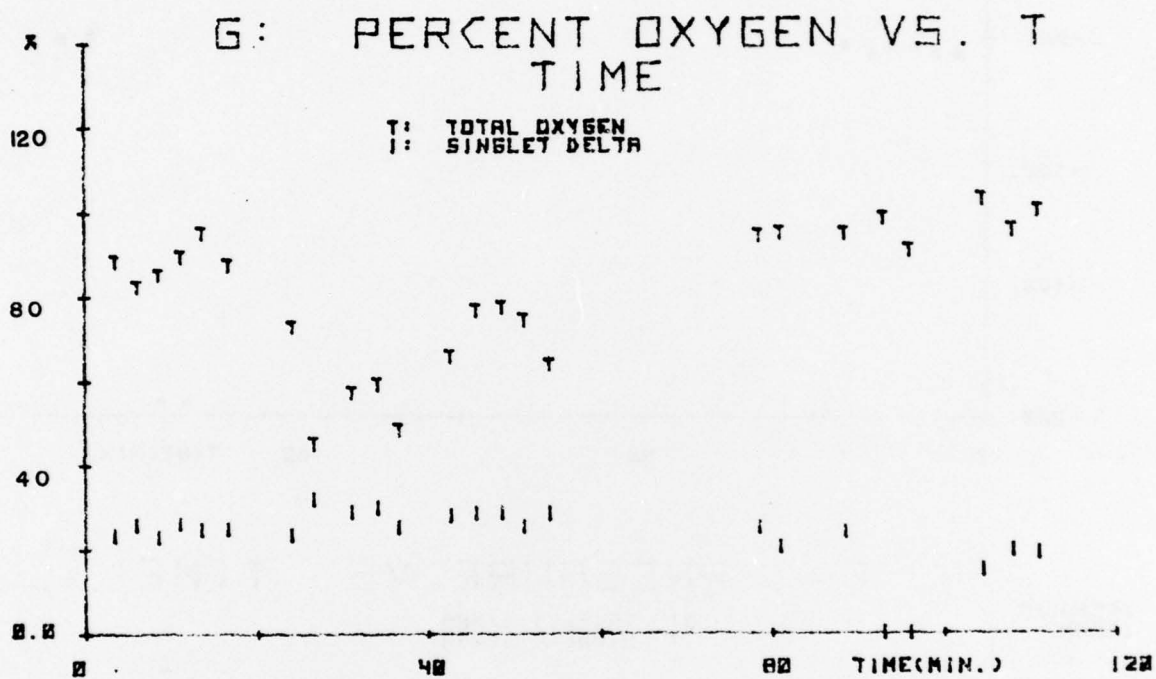


FIG. 11

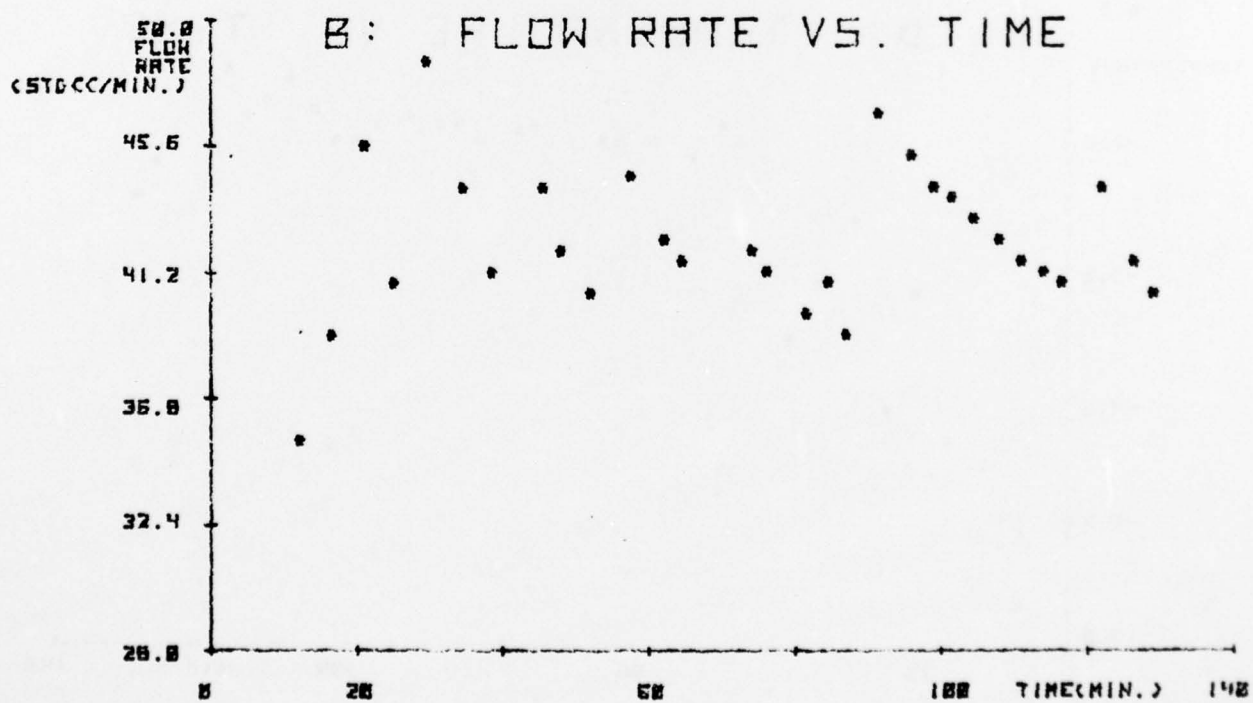
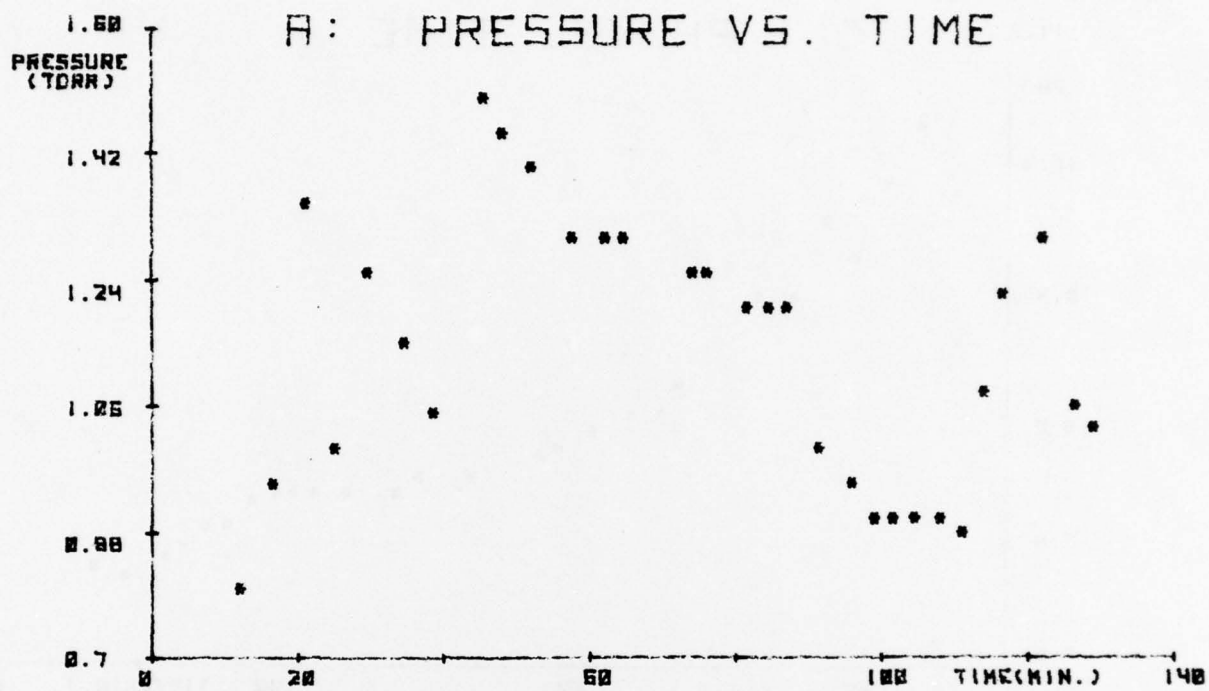


FIG. 11

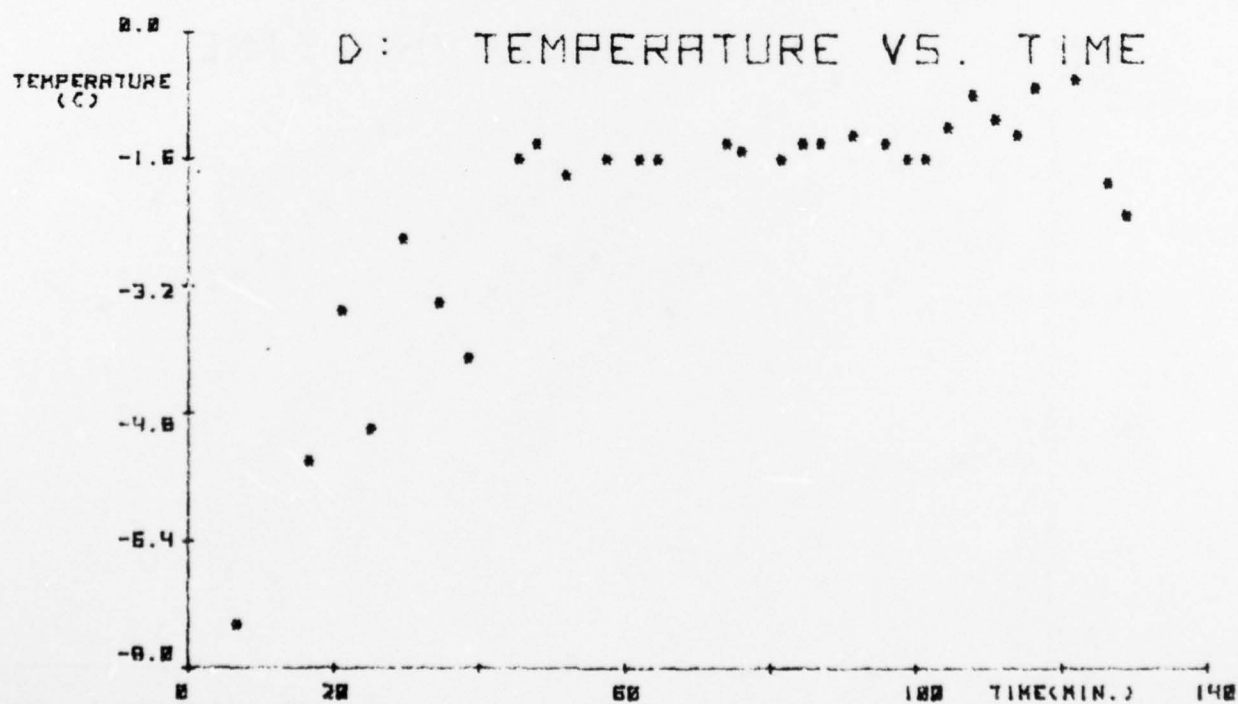
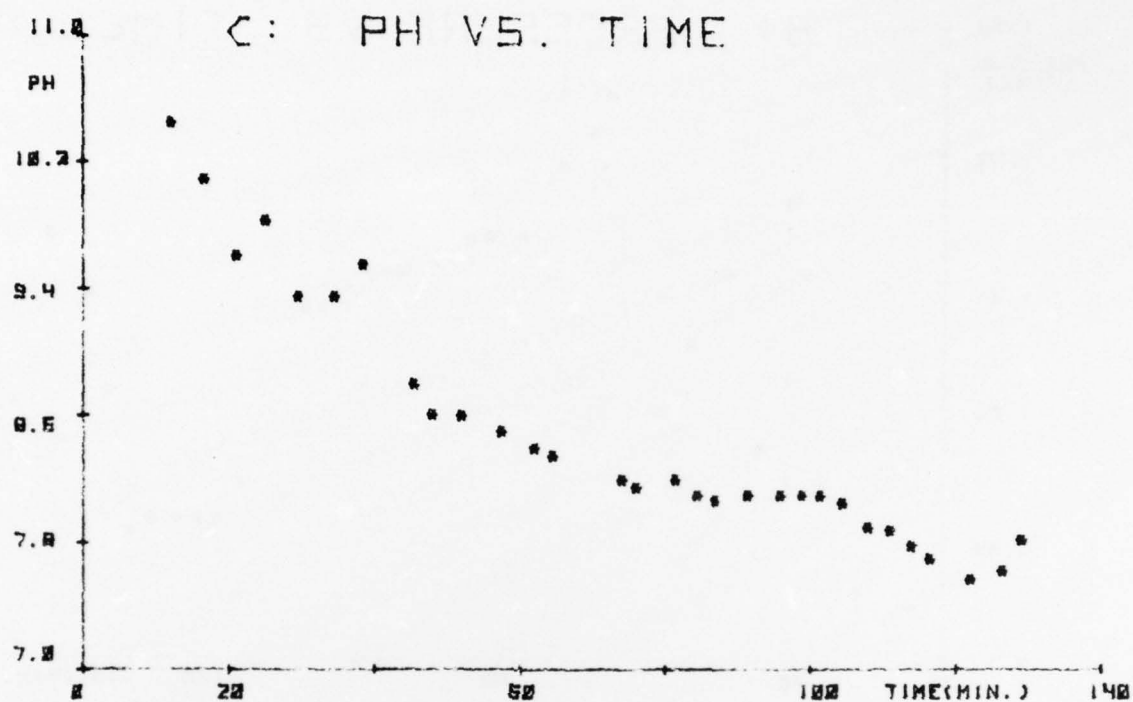


FIG. 11

